

(19)



Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number:

0 399 377
A1

(12)

EUROPEAN PATENT APPLICATION

(21) Application number: 90109375.7

(51) Int. Cl.⁵: C10M 173/00

(22) Date of filing: 17.05.90

(30) Priority: 19.05.89 JP 126375/89

(43) Date of publication of application:
28.11.90 Bulletin 90/48(64) Designated Contracting States:
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(54) Cold rolling oil for steel sheet.

(57) In the cold rolling oils for steel sheets of the present invention, when 0.2 - 5% by weight of a high-molecular nonionic surfactant having a molecular weight of 2000 - 15000 and an HLB value of 5 - 9 as well as 0.2 - 5% by weight of another nonionic surfactant having 12 - 16 HLB value are incorporated with a cold rolling oil as emulsifying and dispersing agents, anti-coalescence of oil particles emulsified and dispersed are remarkably improved, and they are less affected by inclusion of iron powder so that excellent stability with time in an emulsified and dispersed state is obtained as well as that its plate-out is significantly improved. Further, a concentration of the resulting cold rolling oils does not decrease even in case of weak stirring force so that stable performance with time are obtained.

In addition, when 0.1 - 10% by weight of an acetylene glycol nonionic surfactant is further incorporated with the above described cold rolling oils for steel sheets of the invention, adverse effects due to inclusion of iron powder are safely avoided.

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Cold Rolling Oil for Steel Sheets

2. BACKGROUND OF THE INVENTION

(1) Field of the Invention

This invention relates to emulsion type cold rolling oils used in a process for cold rolling of steel sheets.

(2) Description of the Prior Art

In cold rolling of steel sheets, a fluid dispersion (called "coolant liquid") which is prepared by emulsifying and dispersing a cold rolling oil in hot water at a concentration of 1 - 10% is usually employed in a manner of circularly jetting the same for the sake of cooling the heat generated at the time of working steel sheets and supplying lubricating oil to rolling rolls and steel sheets to build it thereon.

Cold rolling oil is a composition obtained by incorporating an oiliness improver, an extreme-pressure additive and an emulsifying agent for emulsifying and dispersing a cold rolling oil with a basic oil such as animal, vegetable and mineral oils, or various synthetic esters and the mixtures thereof obtained from two or more of these basic oil components.

Adhesion (plate-out) of a lubricating oil to steel sheets or rolling rolls is significantly affected by an emulsified and dispersed state of a cold rolling oil. In general, the larger diameter of emulsified and dispersed particles brings about the better plate-out so that lubricity is elevated. Furthermore, in rolling, stability in lubrication is important so that variation in lubricity interfere seriously with rolling operation. However, there is such a tendency that an emulsified and dispersed state of a cold rolling oil varies during storage of the coolant liquid in a coolant tank and circular use thereof, so that it is difficult to maintain a constant emulsified and dispersed state. For this reason, the lubricity varies and it will seriously interfere with working stability.

One of reasons for variation with time in emulsification and dispersion is due to particle size growth as a result of coalescence of dispersed particles of lubricating oil, and another is in that emulsification and dispersibility are affected adversely by inclusion of iron powder produced at the time of rolling and working iron sheets. While a cold rolling oil which has been emulsified and dispersed maintains particles having a comparatively uniform particle diameter which is well-balanced with its stirring condition in early stage of

the dispersion, the particle diameter distributes gradually over a wide range from small to large particle diameters as a result of coalescence and destruction of the particles. Furthermore, as a consequence of inclusion of iron powder, coalescence of dispersed particles occurs, whereby particles having larger particle diameters are produced. Such lubricating oil particles having a large particle diameter float easily in a coolant liquid tank, so that they float or are caught by the coolant liquid dependent upon changes in stirring condition. Thus, a distribution of dispersed particle diameters of the lubricating oil in the coolant liquid to be supplied to rolls or rolling steel sheets fluctuates. As a result, its plate-out changes thereby to bring about variation in lubrication.

In order to avoid the phenomenon as described above, a type, an addition amount and the like of emulsifying and dispersing agents to be incorporated with a cold rolling oil have been studied. Heretofore, a nonionic emulsifier having a molecular weight of 1000 or less has been used as an emulsifying and dispersing agent to be incorporated with a cold rolling oil for steel sheets. Recently, use of water-soluble cationic high-molecular compounds has also been studied and a part of which has been put to practical use for the sake of improving stability with time in respect of an emulsified and dispersed state. However, it is difficult to solve the problems as described above by the use of the nonionic emulsifier as previously mentioned herein. On one hand, while the stability with time is remarkably improved in respect of the emulsified and dispersed state in case where a water-soluble cationic high-molecular compound is used, such stability with time is easily influenced by water quality such as pH, hardness, component and the like of water used because the emulsifier is cationic, on the other hand. Accordingly, water quality control is required and in addition, there arises another problem that since a water-soluble cationic high polymer exhibits no oil solubility, a cold rolling oil becomes two-part liquid system so that its emulsifying and dispersing operability is poor.

In recent years, improvements in operating efficiency are promoted by increase of rolling speed and draft, so that increasingly better lubricity and its stability with time are required for a cold rolling oil. In order to comply with such requirements, elevations in plate-out of a coolant liquid and stability of an emulsified and dispersed state are necessary.

3. OBJECT OF THE INVENTION

It is an object of the present invention to solve the various problems involved in conventional cold rolling oils mentioned above and to provide cold rolling oils which are excellent in lubricity and have good stability with time. Then, efficiency in cold rolling working may be elevated by the use of the cold rolling oils of this invention, whereby it is contemplated to contribute to manufacture of cold-rolled steel sheets.

4. BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a schematic diagram showing a pump circulation tester for evaluating emulsifying and dispersing properties of a cold rolling oil.

5. DESCRIPTION OF THE PREFERRED EMBODIMENTS

The cold rolling oils for steel sheets according to the present invention have been completed on the basis of such discovery that when a specified nonionic surfactant is incorporated with a cold rolling oil, excellent emulsifying and dispersing properties which have never been heretofore can be afforded to the cold rolling oil. More specifically, the present inventors have discovered that when 0.2 - 5% by weight of a high-molecular nonionic surfactant having a molecular weight of 2000 - 15000 and an HLB value of 5 - 9 is incorporated with a cold rolling oil as an emulsifying and dispersing agent, anti-coalescence of oil particles emulsified and dispersed are remarkably improved, and it is less affected by inclusion of iron powder so that stability with time in an emulsified and dispersed state is obtained as well as that its plate-out is significantly improved. However, even if only the above described high-molecular nonionic surfactant is incorporated with a cold rolling oil, a stable floating oil is produced with time so that it decreases a concentration of the cold rolling oil in the case where stirring force is extremely weak. In this respect, it has been found that when 0.2 - 5% by weight of a nonionic surfactant having 12 - 16 HLB value is incorporated with a cold rolling oil, a concentration of the cold rolling oil does not decrease even in case of weak stirring force so that stable performance with time can be obtained.

Because of this finding, excellent cold rolling oils for steel sheets which have never been obtained may be prepared. In addition, as a result of the present inventors' further study, it has been discovered that when 0.1 - 10% by weight of acetylene glycol nonionic surfactant is further incorporated with the above described cold rolling oils

for steel sheets according to the present invention, adverse effects due to inclusion of iron powder can safely be avoided. Hence, separate novel cold rolling oils for steel sheets have been also invented herein.

More specifically, the present first invention relates to a cold rolling oil composition for steel sheets characterized by incorporating 0.2 - 5% by weight of a nonionic surfactant having a molecular weight of 2000 - 15000 and an HLB value of 5 - 9 and 0.2 - 5% by weight of a nonionic surfactant having an HLB value of 12 - 16 with a cold rolling oil containing a basic oil selected from the group consisting of animal oils, vegetable oils, mineral oils, synthetic esters and the mixtures of two or more of them. The present second invention relates to a cold rolling oil composition for steel sheets characterized by further incorporating 0.1 - 10% by weight of an acetylene glycol nonionic surfactant with the cold rolling oil composition for steel sheets according to the first invention.

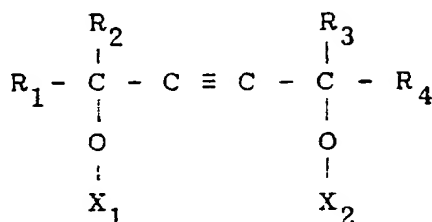
An example of the nonionic surfactants having a molecular weight of 2000 - 15000 and an HLB value of 5 - 9 includes copolymers of propylene glycol and ethylene glycol as well as esters or polyesters prepared from fatty acids, polyfatty acids or polycondensed fatty acids and alcohols such as ethylene glycol, glycerin, sorbitol, sorbitan and the like or polyalcohols. When these surfactants have a molecular weight of less than 2000, effects of anti-coalescence with respect to oil particles are inferior, while when the molecular weight exceeds 15000 so far as the surfactants which were obtained by the present inventors are concerned, oil solubility becomes poor. In case of an HLB value of less than 4 or more than 9, anti-coalescence is poor in either case, besides plate-out is not improved. Further, in the case where an amount of these surfactants to be added is less than 0.2% by weight, effects in anti-coalescence with respect to oil particles are poor, while even if it exceeds 5% by weight, the effects are in saturated conditions so that the higher amount of addition is useless.

Next, an example of nonionic surfactants having an HLB value of 12 - 16 includes polyoxyethylene alcohol ether, polyoxyethylene nonylphenyl ether, polyoxyethylene fatty ester, polyoxyethylene sorbitan ester, polyoxyethylene sorbitol ester and the like.

If an HLB value is less than 12, stability in an emulsified and dispersed state in case of weak agitation cannot be attained, while when the HLB value exceeds 16, oil solubility becomes inferior. An addition amount of the nonionic surfactants of the above described type is sufficient for 0.2% by weight, and even if it exceeds 5% by weight, the effects in addition of the nonionic surfactants reach the saturation. Accordingly, there is no need of the

higher amount of addition.

The acetylene glycol nonionic surfactants have the following general formula:



wherein R_1 and R_4 are H or C_nH_{2n+1} , R_2 and R_3 are H or CH_3 , X_1 and X_2 are H or $(C_2H_4O)_nH$, and n is an integer 1 or more.

In case where an addition count of this type of the nonionic surfactants is less than 0.1% by weight, adverse effect of inclusion of iron powder cannot be suppressed perfectly, while even if it exceeds 10% by weight, the effects of addition of the surfactants are saturated, so that no more addition is needed.

The essential requisite for the cold rolling oils for steel sheets according to the present invention is to contain the above specified nonionic surfactants in the basic oil, but it is to be noted that the cold rolling oils of this invention do not exclude incorporation of additives such as various oiliness improvers, extreme-pressure additives and the like which are generally employed in the art, and further they do not exclude also addition of the other surfactants if required.

As mentioned above, stability with time in emulsified and dispersed state of a cold rolling oil is affected by two major causes, i.e., coalescence of dispersed lubricating oil particles and inclusion of iron powder.

It is known in general that when protecting action with respect to surfaces of dispersed particles is potent, excellent anti-coalescence is attained. Furthermore, particle surfaces of such iron powder produced at the time of rolling steel sheets are lipophilic so that they are easily compatible with lubricating oil particles. As a result, the protecting action with respect to the surfaces of lubricating oil particles is damaged by such compatible iron powder so that it brings about coalescence of lubricating oil particles to produce larger particles containing iron powder. Accordingly, in order to elevate stability with time in emulsified and dispersed state of a cold rolling oil, it is required to make protecting action with respect to surfaces of oil particles more potent to improve the anti-coalescent property and to make the lubricating oil so as not to be easily affected by produced iron powder.

For the sake of elevating anti-coalescent property, it is effective to thicken protective films on the

surfaces of oil particles, and at the same time in order to make the effects attained still stable with time, it is necessary that protective films for oil particles exist stably on the interfaces thereof. Since the nonionic surfactants having a molecular weight of 2000 - 15000 and an HLB value of 5 - 9 used in the present invention are ones having a higher molecular weight than that of nonionic surfactants which have been heretofore employed, the former surfactants can thicken the protective films on the surfaces of oil particles. For this reason, coalescence of oil particles and adsorption of iron powder to the oil particles can be prevented. With respect to HLB value of a nonionic surfactant of the type described above, the reason why a value 5 - 9 is efficient is in that oil solubility is too strong in case where the HLB value is less than 5, while when it exceeds a value 9, water solubility becomes potential. As a result, the surfactant does not stably exist on the interfaces so that stable protective films cannot be obtained on the surfaces of oil particles. Moreover, as to action for improving plate-out, it is considered that such a high molecular weight nonionic surfactant having an HLB value of 5 - 9 produces easily W/O emulsion, and in case of dispersion in hot water, W/O/W emulsion is produced so that plate-out is elevated. Thus, when a nonionic surfactant having a molecular weight of 2000 - 15000 and an HLB value of 5 - 9 is incorporated with a cold rolling oil, a coolant liquid having excellent anti-coalescent property and plate-out can be obtained. However, only this type of high-molecular nonionic surfactant is incorporated, there is such a tendency that the form of a cold rolling oil emulsion shifts from W/O/W emulsion to W/O emulsion in the case where the coolant liquid is weakly agitated. As a result, the stabilized W/O emulsion comes to float on the coolant liquid, and this is not desirable.

Since the W/O emulsion thus produced is hardly dispersed into the coolant liquid, although' distribution of oil particle diameters in the coolant liquid does not change, it results in decrease in concentration of the cold rolling oil. As a countermeasure, a nonionic surfactant having an HLB value of 12 - 16 is further incorporated with the above cold rolling oil, so that production of W/O emulsion is prevented, and a stabilized emulsion form can be obtained.

Next, explanation will be made on acetylene glycol nonionic surfactants. This type of surfactant has a triple bond at the central position of its molecule and OH groups at the positions adjacent thereto, so that the triple bond portion exhibits strong polarity. Because of this polarity, the surfactant is adsorbed on the surfaces of produced iron powder, whereby the surfactant makes the iron powder surfaces hydrophilic. Accordingly, adverse

effects of inclusion of iron powder can perfectly be suppressed by the advantage of addition of the latter nonionic surfactant to the cold rolling oils of the first invention.

Examples

The advantages of the present invention will make clearer hereinbelow by illustrating examples together with comparative examples.

<Surfactants under Test>

Group A ... molecular weight 2000 - 15000, HLB value 5 - 9 (including those being out of the range specified)

Group B ... HLB value 12 - 16 (including those having a value less than 12)

Group C ... Acetylene glycol surfactants

Group D ... Low-molecular weight nonionic surfactants

Group E ... Water-soluble high-molecular nonionic surfactants

A - 1 Pluronic L61, HLB value 5.6, MW 2000

A - 2 Pluronic L121, HLB value 5.0, MW 4500

A - 3 Hypermer A60, HLB value 6.0, MW 15000

A - 4 Hypermer B261, HLB value 8.0, MW 5000

A - 5 Hypermer B246, HLB value 5.5, MW 5000

A - 6 Pluronic L31, HLB value 7.1, MW 1100

A - 7 Pluronic L101, HLB value 4.5, MW 3800

A - 8 Hypermer A409, HLB value 10.0, MW 9000 ("Pluronic" means polyalcohol type surfactants manufactured by Asahi Electrochemical Industries Co., Ltd. and "Hypermer" means ester type surfactants manufactured by ICI Company)

B - 1 polyoxyethylene (20 mol) sorbitan monooleate, HLB value 15.0

B - 2 Polyoxyethylene (9 mol) nonyl-phenyl ether, HLB value 13.0

B - 3 Polyoxyethylene (30 mol) stearate, HLB value 16.0

B - 4 polyoxyethylene (40 mol) sorbitol tetraester, HLB value 12.5

B - 5 Polyoxyethylene (20 mol) sorbitan trioleate, HLB value 11.0

C - 1 2,4,7,9-tetramethyl-decene-4,7-diol

C - 2 Surfactant obtained by adding 4 mol of ethylene oxide to the above material

C - 3 3,6-dimethyl-4-octene-3,6-diol

C - 4 Surfactant obtained by adding 7 mol of ethylene oxide to the above material

D polyoxyethylene (6 mol) nonyl-phenyl ether, HLB value 10.8

E Acetic acid salt of N,N-dimethylamino polymethacrylate (MW 100,000)

<Cold Rolling Oil under Test>

In order to make comparison in various performances easy, such materials are prepared by adding a variety of surfactants to a mixture obtained by adding 3% of stearic acid to tallow, and the materials thus prepared are used for test.

(Examples of the first invention)

Example 1 A - 1 (1%), B - 1 (1%)

Example 2 A - 2 (2%), B - 2 (3%)

Example 3 A - 3 (0.3%), B - 3 (2%)

Example 4 A - 4 (5%), B - 4 (0.2%)

(Examples of the second invention)

Example 5 A - 5 (1%), B - 1 (4%), C - 3 (1%)

Example 6 A - 3 (1%), B - 2 (1%), C - 1 (5%)

Example 7 A - 4 (3%), B - 4 (1%), C - 4 (0.1%)

Example 8 A - 2 (2%), B - 2 (3%), C - 2 (9%)

(Comparative Examples)

Comparative Example 1 A - 3 (0.1%), B - 3 (2%)

Comparative Example 2 A - 6 (2%), B - 4 (3%)

Comparative Example 3 A - 7 (2%), B - 4 (3%)

Comparative Example 4 A - 8 (3%), B - 2 (2%)

Comparative Example 5 A - 7 (3%), B - 3 (4%), C - 2 (0.05%)

Comparative Example 6 A - 4 (3%), B - 1 (0.1%), C - 4 (1%)

Comparative Example 7 A - 4 (3%), B - 5 (1%), C - 3 (2%)

Comparative Example 8 D (3%)

Comparative Example 9 E (2%)

Comparative Example 10 Commercially available tallow cold rolling oil (acid value 5.8, saponification value 196)

<Performance Test>

1. Emulsion and Dispersion Stability

1) Stability with Time

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Emulsifying and dispersing property test was carried out by the use of a pump circulation tester as shown in the Figure of the accompanying drawing. In the testing method, a ratio of the tank capacity for coolant liquid to a circulating amount as well as a stirring method simulated an actual apparatus.

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Conditions:

Coolant liquid, Concentration 3%, Temperature 60 °C, Capacity 30 t, Using ion exchanged water, Circulating amount 4 l/min.

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Testing Method:

After stirring a fresh oil for 3 hours to which was added 1000 ppm of a produced iron powder gathered from a working site, agitation was further continued for 3 hours, and average particle diameter of dispersed oil particles in a spray liquid was investigated as to its variation with time by the use of a coal-tar counter (TA-II type). Furthermore, only the stirrer (at 3 of Figure 1) was stopped after completing the above described test, the circulation was continued for 3 hours to produce a floating oil, thereafter, the stirrer was again operated, and the concentration of a spray liquid after circulating 1 hour was measured.

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		(Average Particle Diameter μm)									
		Example					Comparative Example				
		1	2	3	4	5	6	7	8	9	10
Fresh Oil	1 hour	10.2	9.8	10.6	10.5	9.8	10.6	9.5	9.8	9.8	7.5
	2 hours	10.0	9.6	10.6	10.3	9.5	10.4	9.3	9.6	9.0	7.0
	3 hours	10.0	9.5	10.4	10.4	9.5	10.4	9.2	9.6	9.5	6.5
Adding Iron Powder	1 hour	10.5	9.9	10.8	10.9	9.5	10.4	9.2	9.6	10.3	8.0
	2 hours	11.0	10.3	11.2	10.9	9.6	10.4	9.4	9.6	10.3	10.0
	3 hours	11.0	10.5	11.5	11.3	9.7	10.5	9.5	9.6	10.3	14.5
Conc. After Test X		2.7	2.9	2.8	2.7	2.9	2.8	3.0	3.0	2.2	2.1
Overall Evaluation		O	O	O	O	⊙	⊙	⊙	⊙	X	X

(Evaluation) Excellent ⊙ O Δ X Inferior

2) Influence by Quality of Water Used

A: Ion exchanged water

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B: Water obtained by adjusting pH of ion exchanged water to a value 8 by using NaOH

C: Hard water (total hardness 150 ppm)

The above enumerated water was used as water for dispersion, and a pump circulating test was effected by employing a fresh oil under the same conditions as that of the above-mentioned test for 1 hour, and average particle diameter was confirmed.

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		(Average Particle Diameter μm)																	
		Example								Comparative Example									
		1	2	3	4	5	6	7	8	1	2	3	4	5	6	7	8	9	10
Water used	A	10.2	9.8	10.6	10.5	9.6	10.6	9.5	9.8	10.7	9.5	11.2	7.5	11.3	9.6	9.5	8.5	9.8	7.5
	B	10.3	9.6	10.5	10.6	9.4	10.6	9.4	9.9	10.6	9.4	11.2	7.4	11.5	9.4	10.1	8.2	15.5	7.2
	C	10.4	9.6	10.5	10.4	9.5	10.6	9.2	9.8	11.5	9.7	11.0	8.5	11.5	9.8	9.9	9.6	16.7	8.3
Evaluation		⊙	⊙	⊙	⊙	⊙	⊙	⊙	⊙	○	⊙	⊙	○	⊙	⊙	○	○	×	○

(Evaluation is the same as that defined in the previous test)

2. Plate-out

A steel sheet was sprayed with each testing
cold rolling oil emulsion, and plate-out thereof was
evaluated. Conditions:
Concentration 3%, Temperature 60 °C
Spray flow rate 600 cc/min.
Spray time 0.5 sec.
Temperature of steel sheet 100 °C

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Evaluation

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A pickup of lubricating oil was measured in
accordance with gravimetric method.

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	Example								Comparative Example										
	1	2	3	4	5	6	7	8	1	2	3	4	5	6	7	8	9	10	
Pickup mg./m ²	520	480	500	500	560	490	550	450	470	310	320	340	280	350	370	320	350	300	320

3. Rolling Test

A rolling test was effected by the use of each testing cold rolling oil emulsion to investigate lubricity. Conditions

Emulsion: Concentration 3%, Temperature 60 ° C

Rolling rolls: 530 mmφ

Rolling speed: 1800 mpm

Rolling sheet: SPCCB material 2 x 20 x 850 mm

Draft: 25%

Evaluation

The evaluation was effected in a rolling load per unit width.

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	Example								Comparative Example								
	1	2	3	4	5	6	7	8	1	2	3	4	5	6	7	8	9
Rolling load kg/mm ²	846	848	845	840	847	836	852	846	876	874	872	886	880	875	876	874	884

As indicated in the above test results, the cold rolling oils for steel sheets according to the present invention have excellent emulsion and dispersion stability as well as excellent plate-out so that it may be said that the cold rolling oils of the present invention have also excellent lubricity.

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As described above, the cold rolling oils for steel sheets according to the present invention exhibit excellent emulsion and dispersion stability as well as excellent plate-out due to effects of the specified nonionic surfactants incorporated with the basic oils of the invention. Accordingly, the cold rolling oils for steel sheets according to the present invention have such excellent advantages that they can provide improvement in lubricity and stability of working in cold rolling operation, whereby its working efficiency can be elevated.

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Claims

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1. A cold rolling oil composition comprising a member selected from the group consisting of animal oils, vegetable oils, mineral oils, synthetic esters and the mixtures of two or more of them as a basic oil; 0.2 - 5% by weight of a nonionic surfactant having a molecular weight of 2000 - 15000 and an HLB value of 5 - 9; and 0.2 - 5% by weight of a nonionic surfactant having an HLB value of 12 - 16.

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2. A cold rolling oil composition comprising a member selected from the group consisting of animal oils, vegetable oils, mineral oils, synthetic esters and the mixtures of two or more of them as a basic oil; 0.2 - 5% by weight of a nonionic surfactant having a molecular weight of 2000 - 15000 and an HLB value of 5 - 9; 0.2 - 5% by weight of a nonionic surfactant having an HLB value of 12 - 16; and 0.1 - 10% by weight of an acetylene glycol nonionic surfactant.

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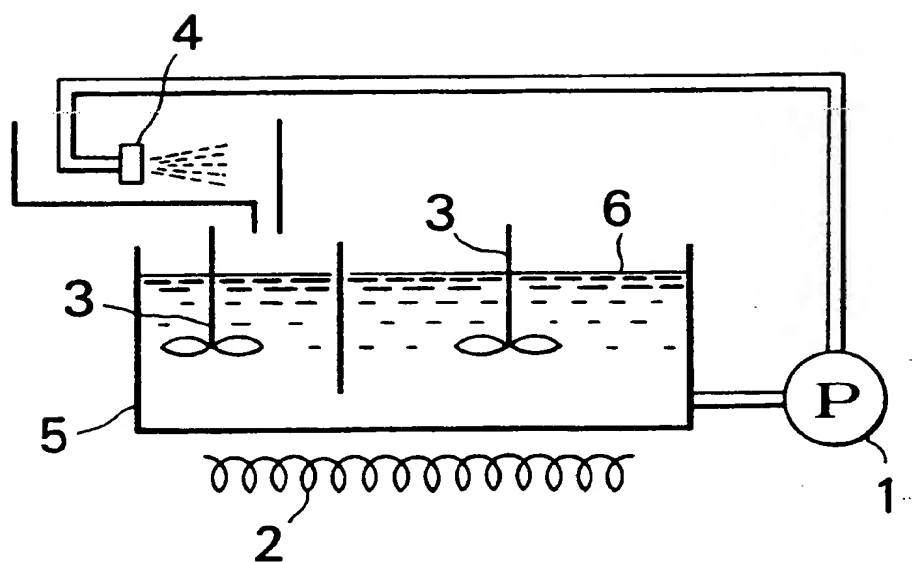


FIG. 1



European Patent
Office

EUROPEAN SEARCH REPORT

Application Number

EP 90 10 9375

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Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
Y	PATENT ABSTRACTS OF JAPAN, vol. 10, no. 67 (C-333)[2124], 15th March 1986; & JP-A-60 203 699 (SHIN NIPPON SEITETSU K.K.) 15-10-1985 ---	1	C 10 M 173/00
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Y	US-A-4 518 512 (KANAMORI) * Whole document *	1	
A	US-A-3 152 990 (COPPOCK et al.) * Whole document *	1	TECHNICAL FIELDS SEARCHED (Int. Cl.5)
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The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 27-03-1990	Examiner DE LA MORINERIE B.M.S.B.
CATEGORY OF CITED DOCUMENTS			
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ----- & : member of the same patent family, corresponding document	

EPD FORM 150 01.82 (P0401)



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The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 27-03-1990	Examiner DE LA MORINERIE B.M.S.E.
CATEGORY OF CITED DOCUMENTS			
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